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PCT  NOTICE INFORMING THE APPLICATION OF THE INTERPRETARY APPLICATION TO THE DESIGNA  (PCT Rule 47.1(c), first see  Date of mailing (day/month/year)  14 September 2000 (14.09.00)	Petentavd. Saks bearb. Sett Eksp.  Alnæs From the INT RNA  Anderson Berg To: PICANEK, van  Doorduin Norsk Hyd o AS  CAN Grunde Norsk Hyd o AS  ERNATIONAL NORVEGE  Howland	TIONAL BUREAU
Applicant's or agent's file reference P9910 International application No.	Besvart (dato)	ORTANT NOTICE
PCT/NO00/00083	08 March 2000 (08.03.00)	iority date (day/month/year) 10 March 1999 (10.03.99)
Applicant NORSK HYDRO ASA et al	1	

 Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice: AU.KP.KR.US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

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 Enclosed with this Notice is a copy of the international application as published by the International Bureau on 14 September 2000 (14.09.00) under No. WO 00/53949

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If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

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If the applicant wishes to proceed with the international application in the **national phase**, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

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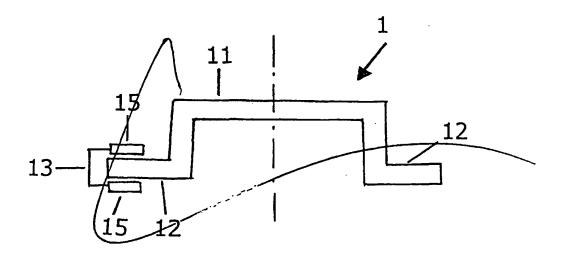
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(54) Title: A FRICTION MEMBER AND A METHOD FOR ITS SURFACE TREATMENT



(57) Abstract

Method of surface treatment of friction PMMC members like brake discs is based on in situ formation of a transfer layer by controlled partial removal of the matrix material from the member's surface.

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WO 00/53949 PCT/NO00/00083

1

## A FRICTION MEMBER AND A METHOD FOR ITS SURFACE TREATMENT

The present invention relates to a method for surface treatment of friction members like brake discs, drums, clutch parts and more particularly to treatment of PMMC based members and further to thereby provided friction members.

Conventional brake discs are presently made of ferrous alloys/cast iron having satisfactory performance and maintaining operative even at substantially elevated temperatures up to above 700°C.

However, the present tendency in the automotive industry to reduce the total weight of vehicles challenges new lighter materials to penetrate also this particular segment of vehicle construction. Furthermore, improved corrosion resistance, as well as wear resistance increasing the lifetime of the friction members up to the expected life period of the vehicles, is also a task when looking for replacement of the present ferrous materials.

Consequently, several patent applications have been filed world-wide recently disclosing use of PMMC (Particle Metal Matrix Composite, e.g. Al-alloy matrix reinforced by ceramic particles) based components applied for different actual applications in vehicles. Shortcomings in common for all these applications based on PMMC base material are the softening phenomena at elevated temperatures, something which results in scoring and even plastic deformation of the members' surface, thus considerably limiting the maximum allowed operating temperatures of the members. Therefore, as a remedy, it is instrumental to provide the basis PMMC-made friction members either with a special composite/ceramic coating layer (thermal spraying of ceramics), or with a transfer surface layer.

WO 00/53949 PCT/NO00/00083

2

The provision of an alternative transfer layer requires the layer to be both stable (adherent to the substrate and reliable) and homogeneous. Furthermore, fast formation of the layer having a sufficient thickness is also requested both from a manufacturing, cost and performance point of view.

One feasible way to cope with the task of increasing the maximum operating temperature is simply to increase the volume percentage of reinforcing particles. Unfortunately, two major disadvantages connected to this "solution", namely increased costs of the PMMC base material and difficulties related to production/casting and especially cutting/machining of the surface, eliminate this as a possibility for a cost efficient manufacturing method.

It is therefore an object of the present invention to provide a novel, fast and cost efficient method of manufacturing friction members avoiding the above mentioned drawbacks and difficulties connected to the hitherto known methods and products.

Another object of the present invention is to provide a fast developing and homogeneous transfer layer exhibiting more stable friction properties, especially at high operating temperatures.

Still another object of the present invention is to provide better protection for the base matrix material against scoring.

These and other objects and features of the present invention are met by provision of a novel manufacturing method of friction members as apparent from claim 1 and the resulting friction member in claim 7, respectively.

The invention will now be described in detail in the following by way of examples of preferred embodiments of the manufacturing method and the resulting members referring to Figures 1-4, where

WO 00/53949 PCT/NO00/00083

3

- Fig. 1 shows in a perspective view a typical disc brake system.
- Fig. 2 illustrates schematically in principle the novel surface topography of the friction member treated in accordance with the present invention,
- Fig. 3 shows a microscope image of an untreated surface, and
- Fig. 4 shows the same surface after exposure to an etching agent as described in the following under Examples.

Referring to Figs. 1 and 2, Fig. 1 shows a disc brake system where the brake disc 1 is the rotating part which together with the friction linings 15 held in place by the caliper 13 creates the friction.

The novel surface treatment according to the invention is applied to the friction surfaces 12 of the disc.

Fig. 2 illustrates schematically a detailed view of the surface of the member (disc) 12 treated in accordance with the present invention.

Contrary to the present practice and trend to add a special surface layer, e.g. in the form of a composite or sprayed ceramic layer, the gist of the present novel treatment method lies in a selective partial removal of the base matrix material from the active to be frictional surface(s) of the member.

The Figure shows in a cross sectional view the (top) surface 2 of the member 1, where the original top layer depicted as 23 has been removed according to the present invention most advantageously by means of chemical etching. This treatment results in a novel surface topography exhibiting a surface with reinforcing (ceramic) particles 22 protruding from the matrix 21, later during the initial break-in activating of the brake system becoming an

WO 00/53949

PCT/NO00/00083

4

integrated part of the transfer layer created through initial wear and material transfer from the lining (pad) material. The resulting increased reinforcement of the transfer layer will provide better protection of the matrix alloy from temperature and shear forces.

Tests conducted on samples of PMMC discs surface treated in accordance with the method confirm formation of a fast developing adherent and homogenous transfer layer exhibiting substantially improved performance characteristics of the treated member.

Furthermore, tests have shown that etching increases the pad wear during the initial use of the disc during the creation of the transfer layer. The degree of etching should therefore be chosen to reach an acceptable initial pad wear. After creation of the transfer layer the actual pad wear falls to a lower level.

## **Examples**

Samples of brake discs made of two different AlSiMg matrix alloys reinforced by SiC particles in an amount of 10 to 30 vol% having a size in a range from 5-30  $\mu$  have been subjected to chemical etching applying a solution of NaOH in concentrations from 5-30% up to 20 minutes.

Comparison to the reference samples based on the measurement of surface roughness, friction and performance at elevated temperatures shows improved characteristics on all measured parameters.

A relatively short etching time proved to be adequate to remove a sufficient amount of the aluminium matrix making the SiC particles to protrude from the surface of the brake disc as illustrated by the attached Figs. 3 and 4 showing sample surfaces before and after the etching treatment according to the present invention, respectively.

The actually applied disc samples were made of AlSiMg alloy added 20 weight% of SiC particles.

The surfaces 2 of the samples 1 were exposed for a period of 2 minutes to 12 weight% water solution of NaOH. (Posting 3 depicts an Al-foil applied on the samples as protection of the surfaces prior to microscopic evaluation of the achieved results).

As clearly manifested in Fig. 4, an exposure time of 2 minutes was sufficient to provide an etched surface 2 with SiC particles 4 protruding from the surface 2.

Generally, an etching time from 1-3 minutes and applying 12.5 weight% NaOH solution at room temperature is apparently sufficient to achieve an adequate degree of etching of the surface. Prolonged etching (in excess of 5 minutes) can result in loosening of SiC particles. The temperature and the control of the flow of the etching agent is decisive for the choice of optimal etching time.

The present invention is not limited to the above described examples of the preferred mode of the surface treatment. Thus also other (similar) methods of surface treatment, e.g. electrochemical pickling or chemical etching by means of an appropriate acid, could be applied without departing from the spirit and scope of the present invention. Thus other alkali-based etching agents than the exemplified NaOH, e.g. KOH, could be applied. Also other types of PMMC material applying other reinforcing particles like Al<sub>2</sub>O<sub>3</sub> instead of the above described SiC-reinforced Al-matrix are the most actual alternatives.

### <u>Claims</u>

- 1. Method of surface treatment of friction members, particularly brake discs/drums or clutch plates in vehicles comprising steps of
  - providing friction members made of PMMC material by any conventional method known per se,
  - in situ formation of a transfer layer on the active surface of the member by removing to a predetermined extent the top layer of the matrix material hereby exposing the surface of the embedded reinforcing particles to a degree providing a transfer layer with increased thickness and stability.
- Method according to claim 1,
   characterized in that
   the in situ formation of the transfer layer is conducted by means of chemical etching of the PMMC material.
- 3. Method according to claim 2,
  characterized in that
  sodium hydroxide (NaOH) in concentrations from 5 to 30% is applied as the etching agent.
- 4. Method according to claim 2,characterized in thatacid reagent is applied as the etching agent.
- Method according to claim 2,
   characterized in that
   KOH is applied as the etching agent.

- 6. Method according to claim 1,
  c h a r a c t e r i z e d i n t h a t
  the in situ formation of the transfer layer is done by electrochemical pickling of
  the PMMC material.
- 7. Friction member, particularly a brake disc/rotor in an automotive vehicle, c h a r a c t e r i z e d i n t h a t the member comprising a PMMC body of Al-alloy reinforced by ceramic particles is provided with a surface topography characterized by surface protruding reinforcing particles, said particles becoming an integrated part of the transfer layer.
- 8. Member according to claim 7,characterized in thatthe Al-alloy is an AlSi alloy reinforced by SiC particles.

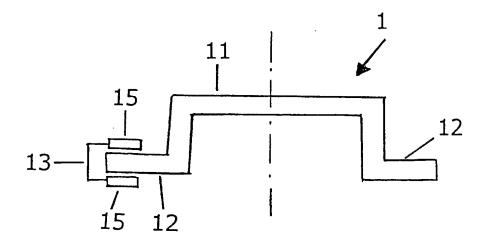


Fig. 1

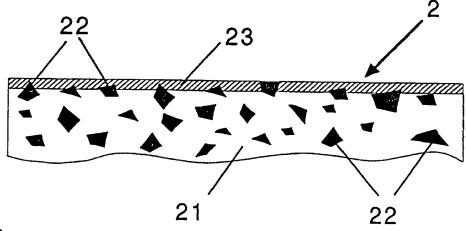
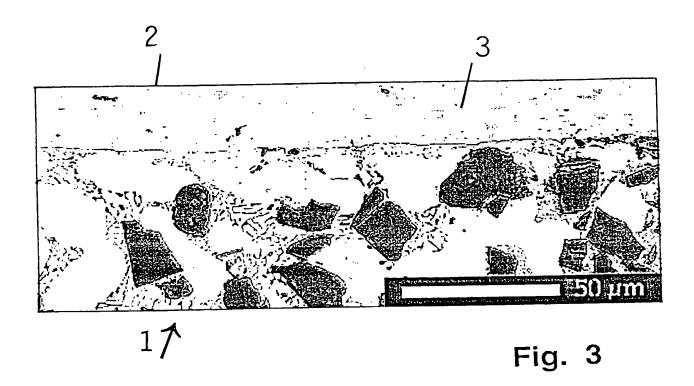


Fig. 2



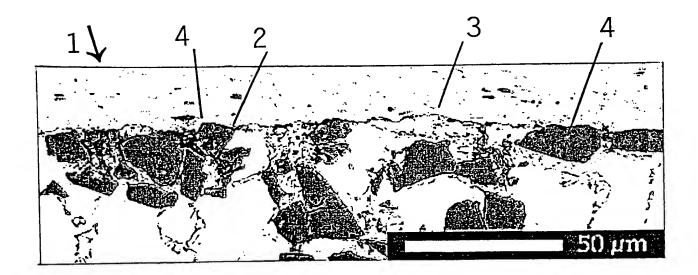


Fig. 4



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# A. CLASSIFICATION OF SUBJECT MATTER IPC7: F16D 69/02, C23C 12/00 According to International Patent Classification (IPC) or to both national classification and IPC **B. FIELDS SEARCHED** Minimum documentation searched (classification system followed by classification symbols) IPC7: F16D, C23C Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched SE, DK, FI, NO classes as above Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPODOC, WPI C. DOCUMENTS CONSIDERED TO BE RELEVANT Category\* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. WO 9205292 A1 (MURPHY, MARTIN, JOHN, MICHAEL), 1,6-82 April 1992 (02.04.92), figure 2, claims 1,2,4-6, 10,12 WO 9726465 A1 (FERODO BREMSBELÄGE TECHNIK-ZENTRUM Α 1,8 GMBH), 24 July 1997 (24.07.97), claims 1-5,10 Further documents are listed in the continuation of Box C. See patent family annex. Special categories of cited documents: later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention document defining the general state of the art which is not considered to be of particular relevance crlier document but published on or after the international filing date document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other step when the document is taken alone special reason (as specified) document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is document referring to an oral disclosure, use, exhibition or other combined with one or more other such documents, such combination being obvious to a person skilled in the art document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 0 6 -07- 2000 <u>16 June 2000</u> Name and mailing address of the ISA Authorized officer Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Igor Gazdik/AB Facsimile No. +46 8 666 02 86 Telephone No. + 46 8 782 25 00



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# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

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International application No.  PCT/NO00/00083  International filing date (day/month/year)  08/03/2000  International Patent Classification (IPC) or national classification and IPC  Priority date (day/month/year)  10/03/1999  International Patent Classification (IPC) or national classification and IPC  International Patent Classification (IPC) or national classification and IPC  International Patent Classification (IPC) or national classification and IPC  International Preliminary examination report has been prepared by this International Preliminary Examining Authoral and is transmitted to the applicant according to Article 36.  This REPORT consists of a total of 4 sheets, including this cover sheet.  This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).  These annexes consist of a total of sheets.	Applicant's or agent's file reference			FOR FURTHER ACTION		otification of Transmittal of International
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Date of submission of the demand  Date of completion of this report  09.02.2001  Date of completion of this report  09.02.2001  Authorized officer  European Patent Office D-80298 Munich			Basis of the report Priority Non-establishment of Lack of unity of inve Reasoned statement citations and explant Certain documents Certain defects in the	of opinion with regard to novelty, ntion t under Article 35(2) with regard ations suporting such statement cited e international application		
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reliminary examining authority:  European Patent Office  D-80298 Munich  Krysta. D	25/09/20	00		09.02	2.2001	
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Fax: +49 89 2399 - 4465 Telephone No. +49 89 2309 2042	ارو	Tel.	+49 89 2399 - 0 Tx: 523	656 epmu d	na, D	La Company

Telephone No. +49 89 2399 2949

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/NO00/00083

<ol> <li>Basis of the report</li> </ol>	l.	Bas	is o	f the	repo	rt
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1.	res <sub>i</sub> the	This report has been drawn on the basis of (substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).):  Description, pages:							
	1-5		as originally filed						
	Cla	ims, No.:							
	1-8		as originally filed						
	Dra	wings, sheets:							
	1/2-	2/2	as originally filed						
2.	With regard to the <b>language</b> , all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.								
	These elements were available or furnished to this Authority in the following language: , which is:								
	the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).								
		the language of pu	blication of the international application (under Rule 48.3(b)).						
		the language of a 155.2 and/or 55.3).	translation furnished for the purposes of international preliminary examination (under Rule						
3.	With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:								
	□ contained in the international application in written form.								
		filed together with the international application in computer readable form.							
		furnished subsequ	ently to this Authority in written form.						
		furnished subsequ	ently to this Authority in computer readable form.						
		☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.							
		The statement that listing has been fu	t the information recorded in computer readable form is identical to the written sequence rnished.						
4.	The	amendments have	resulted in the cancellation of:						
		the description,	pages:						
		the claims,	Nos.:						

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/NO00/00083

	_	Also a discovida asa	-1			
		the drawings,	sheets:			
5.	5. This report has been established as if (some of) the amendments had not been made, since they have be considered to go beyond the disclosure as filed (Rule 70.2(c)):					
		(Any replacement she report.)	eet contai	ning such	amendments must be referred to under item 1 and annexed to this	
6.	Add	litional observations, if	necessar	y:		
V.		soned statement und tions and explanation			ith regard to novelty, inventive step or industrial applicability;	
1.	Stat	ement				
	Nov	relty (N)	Yes: No:	Claims Claims	1-8	
	Inve	entive step (IS)	Yes: No:	Claims Claims	1-8	
	Indu	ıstrial applicability (IA)	Yes: No:	Claims Claims	1-8	

2. Citations and explanations see separate sheet

# VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted: see separate sheet

# INTERNATIONAL PRELIMINARY

International application No. PCT/NO00/00083

**EXAMINATION REPORT - SEPARATE SHEET** 

## Point V:

### State of the art: 1.

An Al-alloy matrix brake disc reinforced by ceramic particles comprising a protective coating as disclosed e.g. in document WO92/05292 (=D1).

#### 2. Object of the invention:

Reduction of the manufacturing costs, in particular costs caused by applying the protective layer.

#### 3. Solution:

According to independent method claim 1 a predetermined extend of the top layer of the matrix material is removed hereby exposing embedded reinforcing particles. This allows in situ formation of a protective transfer layer.

According to independent device claim 7 the brake disc body is characterized by surface protruding reinforcing particles. As in claim 1 said protruding particles allow in situ formation of a protective transfer layer whereby said particles become an integrated part of the transfer layer.

Allowing in situ formation of a protective transfer layer by providing reinforcing particles protruding from the body surface is neither known from nor suggested by the available state of the art. Therefore, independent claims 1 and 7 and the following dependent claims are regarded as being new and inventive.

### **Point VII:**

A document (e.g. D1) reflecting the state of the art as described in the application 1. should have been cited (Rule 5 PCT).

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# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

	or agent's	file reference	FOR FURTHER AC	TIAN -	See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416
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nternationa	• •		International filing date (da 08/03/2000	ay/montn/yea	ar) Priority date (day/month/year) 10/03/1999
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NORSK	HYDRO	ASA et al.			
			nination report has been paccording to Article 36.	prepared by	this International Preliminary Examining Author
2. This f	REPORT	consists of a total of	of 4 sheets, including this	cover shee	et.
b	een ame	nded and are the ba		sheets cont	lescription, claims and/or drawings which have taining rectifications made before this Authority under the PCT).
These	annexe	s consist of a total o	of sheets.		
3. This r	eport co	ntains indications re	lating to the following item	ıs:	
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III IV V VI VIII Date of sub	□ No □ La ⊠ Ro cit □ Co □ Co □ Co □ mailing ac examining Europea D-80298	iority on-establishment of ack of unity of invented statement of attorns and explanate ertain documents ciertain defects in the ertain observations of the demand	under Article 35(2) with re- tions suporting such states ited international application on the international applica	gard to nov ment ation  Date of com	relty, inventive step or industrial applicability;  appletion of this report

International application No. PCT/NO00/00083

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٧.		soned statement unditions and explanatio			with regard to novelty, inventive step or industrial applicability; ach statement	
1.	State	ement				
	Nov	elty (N)	Yes: No:	Claims Claims	· -	
	Inve	ntive step (IS)	Yes: No:	Claims Claims		
	Indu	strial applicability (IA)	Yes: No:	Claims Claims		

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